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AD-A265 016

ARMY RESEARCH LABORATORY

Dry In-Line Thermoplastic Matrix Impregnation, Phase I - SBIR

L. Scot Duncan

ARL-CR-55

April 1993

prepared by

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under contract

DAAL04-92-C-0011

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REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	April 1993	Final Report - August 1992 - January 1993

4. TITLE AND SUBTITLE
Dry In-Line Thermoplastic Matrix Impregnation
Phase I - SBIR

5. FUNDING NUMBERS
Contract No.
DAAL04-92-C-0011

6. AUTHOR(S)
L. Scot Duncan

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
North Coast Innovation, Inc.
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Cleveland, OH 44125

8. PERFORMING ORGANIZATION REPORT NUMBER
NCI/93-07

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
U.S. Army Research Laboratory
Watertown, MA 02172-0001
ATTN: AMSRL-OP-PR-WT

10. SPONSORING/MONITORING AGENCY REPORT NUMBER
ARL-CR-55

11. SUPPLEMENTARY NOTES
Seth Ghiorse, COR

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution unlimited.

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

The objective of this program is to develop an in-line thermoplastic impregnation module which can be used with existing automated manufacturing systems, such as filament winding equipment, which were originally designed for use with thermoset materials. During Phase I, the ability to melt impregnate non-woven glass tow with polyphenylene sulfide (PPS), nylon, and polycarbonate resins has been demonstrated at the breadboard level by using a laboratory injection molding press to feed resin to a melt coating module. The tow was preheated prior to coating by means of a convection heating tunnel. Key parameters governing melt penetration included incoming tow temperature and melt viscosity. Further development of the process would allow the creation of composite shapes and structures which could be post processed by thermoforming techniques and would have the desirable performance characteristics of thermoplastics such as damage tolerance (impact resistance) and repairability.

14. SUBJECT TERMS
Thermoplastics, Composites, Fibers, Pultrusions, Impregnation, Manufacturing, Filament winding

15. NUMBER OF PAGES
46

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
Unclassified	Unclassified	Unclassified	UL

NSN 7540-01-280-5500

Standard Form 298 (Rev. 2-89)
Prescribed by ANSI Std. Z39-18
298-102

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I. SUMMARY

This program was undertaken under SBIR Contract DAAL04-92-C-0011 sponsored by the Army Materials Technology Laboratory, Watertown, MA, to explore the feasibility of developing a dry in-line impregnation module for use with thermoplastic resins and glass or carbon fibers on automated composites fabricating systems.

During Phase I, a simple bench top impregnation module was designed, built and demonstrated. Samples were run with polyethylene, polycarbonate, nylon, polyphenylene sulfide (PPS), and polyether ether ketone (PEEK). All materials, except PEEK, were processed with some degree of success. The majority of the experimental work was done with PPS which proved to have excellent processing characteristics for this process as well as good mechanical properties at high temperatures.

The sample thermoplastic composite materials produced were examined for complete melt penetration and for fiber content by sectioning the coated tow, polishing the samples, and examining them visually under a microscope.

The initial testing showed that good penetration could be obtained if the tow was sufficiently preheated. Fiber content was somewhat below that obtained by other processing methods, such as press lamination, but was still sufficiently high for many applications. Due to a limited preheater length in the breadboard test rig, processing speed was limited to less than 60 inches per minute. With a more effective means of preheating the tow, much higher processing speeds would be possible. Such higher speeds would be required to make the process commercially viable.

The next step in the development of the in-line impregnation module should be to design and build a prototype unit which could operate on line with a filament winder in a continuous fashion. The basic concept demonstrated here can be used on-line at speeds of several inches per second if it is upgraded with a continuous melt supply system and a multi-loop tow preheater. A multitow system, processing about six tows and outputting a ribbon shape is envisioned.

Various potential Army end users of the technology, including The Tank and Automotive (TACOM), The Missile Command (MICOM), The Natick Research, Development and Engineering Center (NRDEC), and The Corps of Engineers Construction Engineering Research Lab (CERL), were contacted to determine their level of interest. All groups contacted expressed a general interest in thermoplastic composites.

The most promising applications identified in initial contacts included various filament wound structures at MICOM and CERL. MICOM is interested in thermoplastic composite rocket motor housings and also as a damage tolerant container structure which would fail by melting (rather than exploding) in case of fire.

CERL has ongoing SBIR efforts in recycled plastics for structure applications. These could, potentially, utilize the in-line module to produce composites employing recycled thermoplastics.

II. INTRODUCTION

Composite materials have played an important role in improving the cost/performance of military hardware for the past two decades. Compared to metal structures, they offer higher strength to weight ratios and lower manufacturing costs in many applications. Until recently, military composites employed thermosetting resin matrix systems almost exclusively. Typical fabrication procedures for thermoset composites involve:

- o Production of prepreg material (woven or nonwoven fibers saturated with partially cured resin)
- o Cutting and hand layup of multiple plies of prepreg
- o Vacuum bagging and autoclaving or compression press consolidation of the layup to achieve the desired density and physical properties.

Studies have shown that, due to the labor intensiveness of the processing, more than 70% of the total cost of an aerospace composite part is process related.[1] Cost comparisons of alternate fabrication methods, like the one in Exhibit I, have shown the clear cut cost advantage of mechanized manufacturing methods, such as filament winding.[2] With the development of computer-controlled winding equipment, even relatively complex shapes can be produced by automated manufacturing methods.

In the future, thermosets are likely to be replaced by thermoplastics in many applications because of performance and fabrication cost advantages. Unfortunately, the changeover will require major changes in manufacturing technology. A recent study by the National Material Advisory Board has identified the development of the required thermoplastic composite processing technology as critically important.[2]

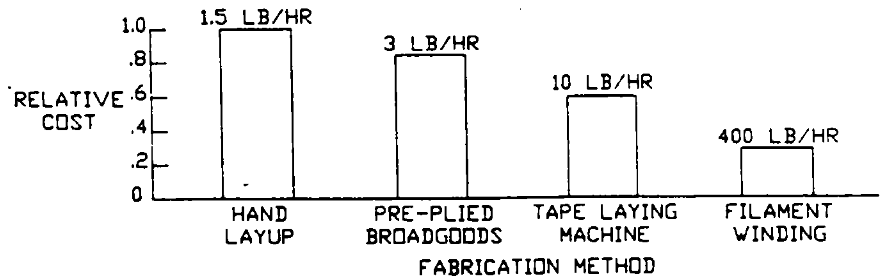


EXHIBIT I - COST ADVANTAGE OF COMPOSITE AUTOMATION
(from ref 2 - National Materials Advisory Board Study)

Exhibit I

A key technology requirement for the commercialization of thermoplastic composites is the development of a simple, solventless approach for impregnating various fiber types with thermoplastic resins which can be placed in-line with existing process equipment. The availability of a dry in-line thermoplastic matrix impregnation module would allow the use of traditional filament winding, pultrusion, and prepreg technology for the fabrication of thermoplastic composite components.

During the planning of this program, a number of alternate impregnation technologies were examined before the melt impregnation approach was selected for further development. Alternatives considered included:

- o Powder coating
- o Dry lamination
- o Melt impregnation

Melt impregnation was selected as the approach for in-line applications because it can be simple, low in cost, and compatable with in-line operation. A number of other technologies are in commercial use today for the production of thermoplastic prepreg which can be processed by traditional (thermoset) layup techniques. Exhibit II presents an overview of the thermoplastic options which were considered before selecting the melt impregnation approach.

The majority of the manhours on this project were devoted to actual laboratory work with a benchtop impregnation system. This "hands on" approach allowed empirical development of a feel for the processing parameters required to later develop a successful impregnation system which could be used in-line on a filament winder.

To minimize the design time, and to avoid the necessity of purchasing substantial amounts of commercial hardware, a laboratory sized injection molding press was borrowed for the duration of the project. The molding press served as a premelter and pressurized supply for the thermoplastic resins. The molding press was fitted with an impregnation block in which the actual impregnation took place. The tow was first preheated and then fed through the impregnation block. After coating, the tow was pulled from the impregnation block by a constant speed mechanical drive. Limitations on the charge size, inherent in the design of the press, restricted operation to between 10 and 20 feet of coated tow per batch. A detailed description of the coating hardware is presented in Section III of the report.

DRY IN-LINE THERMOPLASTIC IMPREGNATION OPTIONS

PROCESS	DESCRIPTION	ADVANTAGES	DISADVANTAGES
WET SYSTEMS	USES SOLVENT, SLURRY OR EMULSION APPROACH TO DISPERSE RESIN THROUGHOUT FIBERS	- MATURE TECHNOLOGIES	- MUST BE DRIED - SUBJECT TO VOIDS - POLLUTION/HEALTH HAZARDS
COMMINGLED STRUCTURES	MONOFILAMENTS OF RESIN ARE COMBINED WITH STRUCTURAL FIBERS IN WOVEN OR NON-WOVEN FORMAT	- EASY HAND LAYUP - REPLACE PREPREG DIRECTLY	- HARD TO CONSOLIDATE - HIGH COST
SINTERED POWDER COATING	FIBERS ARE POWDER COATED AND SINTERED TO CREATE A FLEXIBLE FORMAT	- EASY HAND LAYUP - REPLACE PREPREG DIRECTLY	- SLOW COATING PROCESS - HARD TO CONSOLIDATE
POWDER COATING WITH TOW CONSOLIDATION	FIBERS ARE POWDER COATED AND PRECONSOLIDATED IN A PULTRUSION-TYPE OPERATION	- FINAL CONSOLIDATION IS FASTER	- SLOW COATING PROCESS - NO PREPREG DRAPE
MELT IMPREGNATION	A CROSS EXTRUDER IS USED TO FORCE MELTED RESIN BETWEEN THE (PREHEATED) FIBERS	- SIMPLE - FAST - MATURE TECHNOLOGY	- MAY DAMAGE FIBERS - NO PREPREG DRAPE
DRY LAMINATION	PREHEATED FIBERS ARE ROLLED INTO PREHEATED RESIN TAPES	- SIMPLE - MINIMIZES FIBER DAMAGE	- REQUIRED PRE-EXTRUDED TAPE - NO PREPREG DRAPE
EXTRUSION/LAMINATION	COMBINES DRY LAMINATION CONCEPT WITH IN-LINE EXTRUDER TO MINIMIZE	- FAST - MINIMIZES FIBER DAMAGE - AUTOMATION COMPATIBLE	- MORE MECHANISM THAN MELT IMPREGNATION

prepared by NORTH COAST INNOVATION, INC.

EXHIBIT II

III. METHODS AND PROCEDURES

The primary focus during this Phase I activity has been to gain some "hands-on" experience with thermoplastic melt impregnation. The long range objective is the development of an in-line impregnation module which can be used with filament winding systems or other automated composite manufacturing device.

Short term objectives included:

- Develop a way to preheat glass or carbon tows of various weights
- Adapt a resin melt supply system to the application on a temporary basis
- Develop a melt impregnation block
- Develop a constant speed take-up system

Benchtop System Hardware Description

Because the program budget was limited by the SBIR Phase I guidelines, equipment was assembled which would be sufficiently versatile for the initial feasibility trials, but, intentionally, did not include all of the instrumentation and control refinements which would be required for full scale prototype development.

An injection molding press was borrowed (Simplomatic Air Ram). This unit utilizes shop air to power the injection ram. The operating statistics for the press are as follows:

- It has a 9/16 inch diameter injection ram directly connected to a 4.0 inch diameter air cylinder, yielding a 50:1 ratio between the injection pressure and the shop supply air. This provides 5000 psi injection pressure at 100 psi shop air pressure. The ram moves in a vertical direction.
- The ram stroke is about 2.5 inches, yielding a theoretical shot volume of 0.62 cubic inches. At a 1.4 resin specific gravity, this equates to a 1/2 oz. shot. In practice, bypass leakage was found to reduce the actual quantity of material delivered to less than this amount by an estimated 10 to 20 percent.
- The press has a thermal system consisting of a 300 watt barrel heater controlled by a Love analog (on/off) control capable of controlling up to 700°F. A secondary analog temperature gage is also provided.

o The press is designed to take a standard size mold block which is 2 inches high by 3 inches wide by 1.375 inches thick. The press nozzle feeds resin to the center top of this mold when the mold is properly installed in the mold clamp.

Starting with the Simplomatic as the heart of the system, the necessary hardware to allow the system to function as a cross head extruder was then designed. The overall hardware layout is shown in Exhibit III and is described in the following paragraphs. Detail drawings are included in Appendix A.

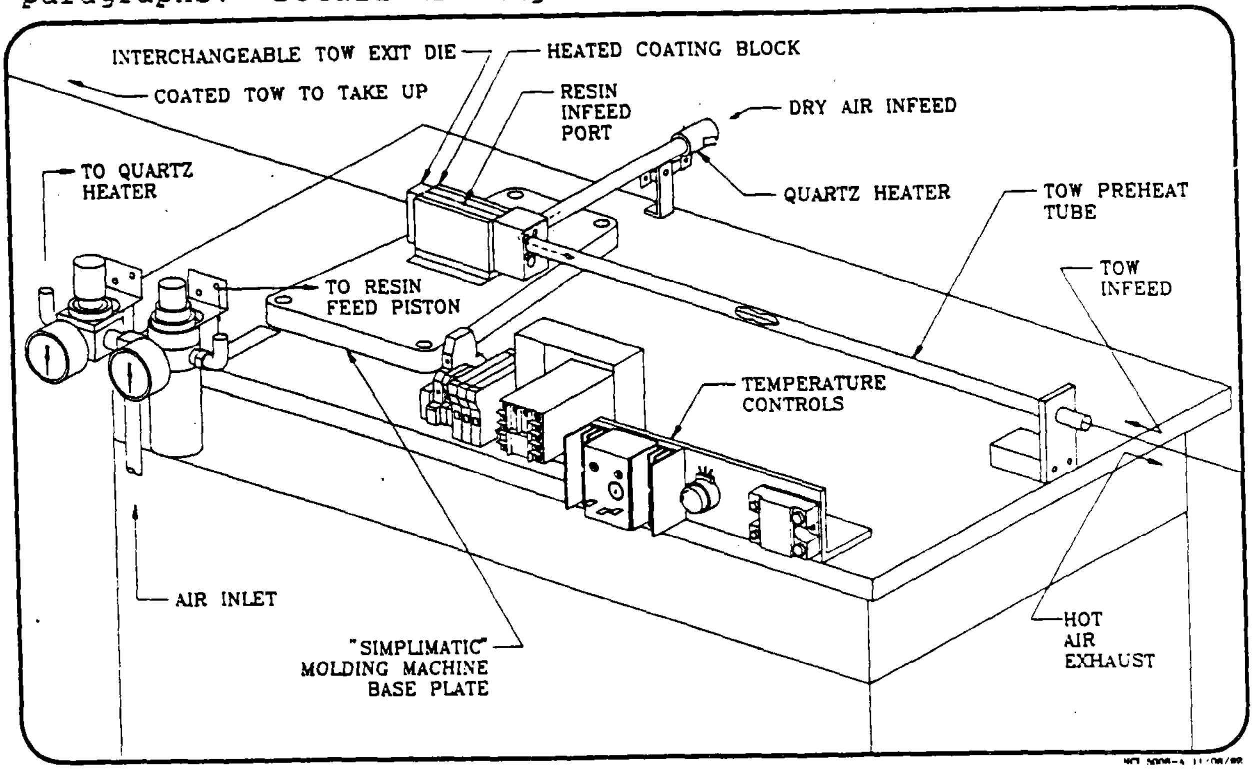


EXHIBIT III - OVERALL HARDWARE LAYOUT

The heart of the system is the Coating Block, as shown in perspective view in Exhibits IV and V. The block is sized to fit into the mold clamp of the Simplomatic press. To keep the coating block hot, it is insulated from the press frame by means of 1/4 inch thick composite glass insulating pads. The operation details are as follows:

o The uncoated tow enters the resin cavity through a relatively long passage. When the fiber is moving, no resin flows out in the upstream direction.

o Resin from the injection ram enters the top of the block in the same manner that a mold is normally filled. The resin fills the coating cavity, surrounding the tow.

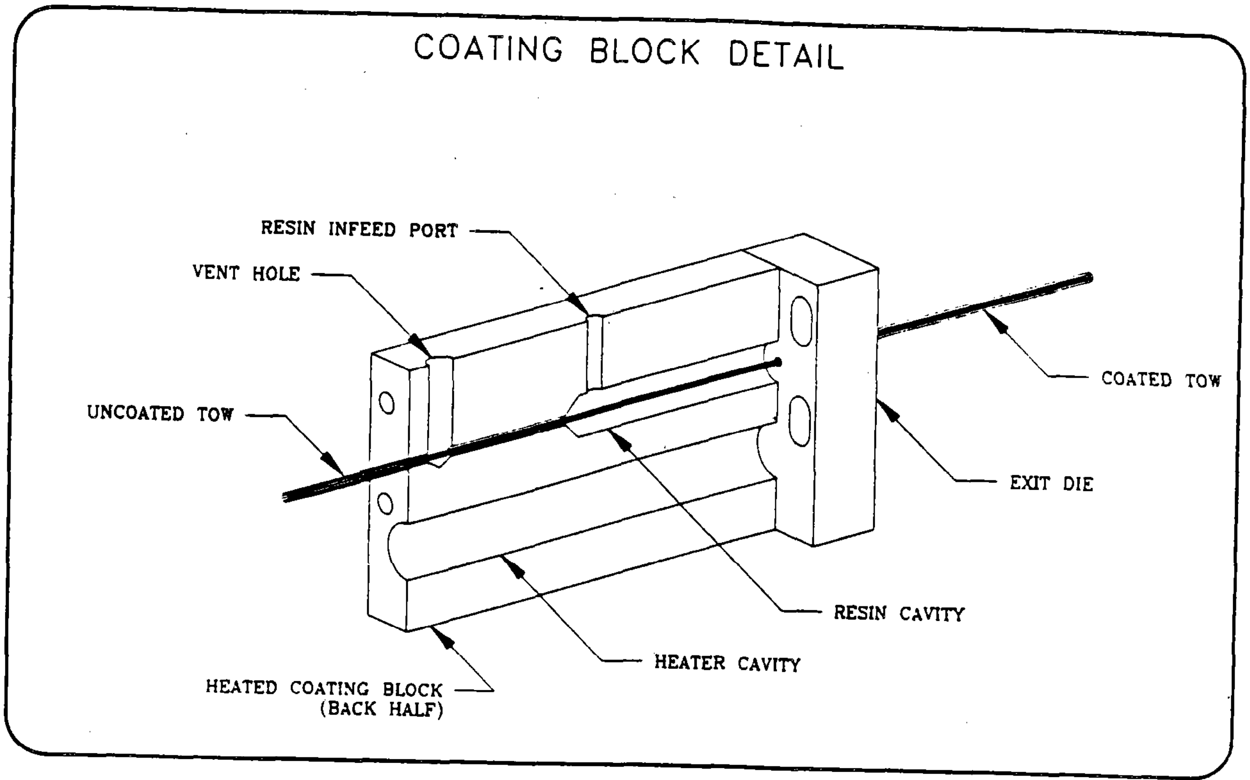


EXHIBIT IV

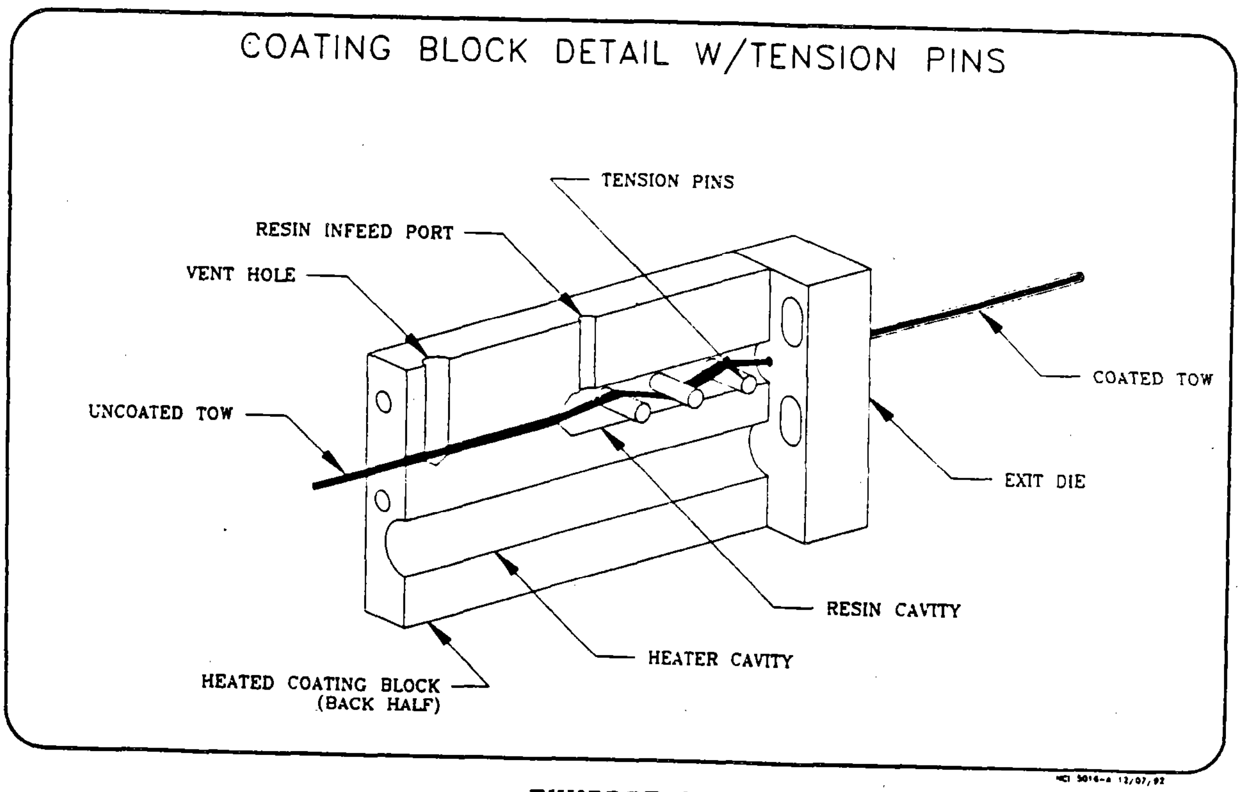


EXHIBIT V

- The coated tow exits through a smaller and shorter orifice in a removable exit die.

- Exhibit V shows the tension pin installation. These devices proved to be very effective in improving resin penetration.

- Temperature in the coating block is independently controlled from the press temperature by means of a cylindrical cartridge heater located in a cavity below the resin cavity. This heater is also 300 watts and is controlled with a Watlow digital controller. The sensing thermocouple is located on the exit die.

The second major subsystem of the benchtop test rig is the tow preheater. This subsystem, also shown in Exhibit III, consists of a Quartz convective heater, a manifold block and a 48 inch length of .500 in. OD by 0.065 in. wall stainless steel tubing. Compressed air is fed through the heater and flows in a counterflow direction to the incoming tow. Thermocouples located at the manifold block and at the midpoint of the tube provided an indication of the air temperature surrounding the incoming tow. The preheater is controlled in an open loop fashion with a solid state current limiting controller.

The final subsystem in the benchtop test rig is the tow take-up system. An existing stepper motor drive was adapted to the task by adding a 10:1 gear reduction and a take-up spool assembly. The resultant system was capable of exerting over 100 pounds of tension at a programmable constant speed (or any desired speed profile). In operation, the takeup was located about 20 feet from the injection press and a steel cable was used to connect the end of the tow to the takeup spool. The coated tow was allowed to cool in a tensioned manner and samples were then cut from the coated tow after it cooled.

<u>Testing Procedures</u>

Since the primary objective of the initial testing was to develop a qualitative feel for the feasibility of melt impregnation with various thermoplastic resins, the test runs were conducted with the objective of "bracketing" the most promising operating conditions. While records were kept of all operating conditions, collecting large amounts of quantitative data was avoided as this data would be test rig specific and would be of little use to future development.

The test procedures included selecting a resin and running at a series of operating conditions while observing qualitatively how the system responded. With some operating experience, it was possible to quickly arrive at a set of operating conditions which produced the best results for each resin type. Samples were collected and examined. A 30X microscope examination of knife cut sections was used during the run to get a quick look at the degree of wetting being obtained and some samples were mounted in epoxy resin and polished at the end of each run.

The major test variables included the following:

- o Adjustable operating temperatures

  - Molding press barrel temperature - Controlled with the press temperature control

  - Coating block temperature - Controlled with the Watlow controller which was connected to the coating block heater

  - Tow preheater inlet and outlet air temperatures - Controlled jointly by varying the quartz heater current and the air flow.

- o Melt supply pressure - Controlled by means of a pressure regulator on the air supply to the press ram cylinder.

- o Tow feed rate - Programmable via the stepper motor drive on the takeup system.

A typical run with a new material consisted of setting probable operating conditions for the material based on known melt temperatures. The press temperature was set for the suggested molding temperature, the coating block temperature was set slightly lower, and the melt supply pressure was gradually increased until some material flow was observed at the outlet and through the coating block vent. Typically, the tow preheater was run with a 700°F air inlet temperature and a 400°F air outlet temperature. Since the preheater was relatively short, the resultant tow temperature was highly dependent on the tow speed and the residence time at start-up and shut-down. In general, the goal was to achieve a tow temperature at or above the melt temperature of the thermoplastic resin. However, it is believe that this was only achieved at low processing speeds.

<u>Preparation of Microscope Samples</u>

To examine the degree of wet-out and resin penetration it was necessary to find a method for visually looking at the cross section of the impregnated tow. Attempts to simply knife cut the tow after coating resulted in a fractured surface. This made it difficult to evaluate which voids were caused by the fracture and which voids were caused by incomplete impregnation. To get away from the fracture problem, short sections were mounted in epoxy resin and polished.

A support fixture was built to hold short lengths of impregnated tow on end in a one ounce cup while resin was poured around them and allowed to cure. Six to eight samples were mounted in a single one ounce cup. A pigment was added to the epoxy resin to provide a contrasting background color in the finished samples.

Once the sample cured, both ends were ground off to make a square-ended cylinder. One end was then wet sanded with successively finer grits of paper, starting with 120 grit and ending with 1500 grit. The finished samples were examined visually with a 30X microscope to determine void content and fiber/resin distributions. Some representative samples were also photographed at 50X and 100X, using an outside metallurgical laboratory.

IV. RESULTS AND DISCUSSION

The benchtop test rig was tested with a representative variety of materials ranging from polyethylene (PE) to polyether ether ketone (PEEK). As was expected, the results varied by material. The best success with a "high performance" engineering material was obtained with polyphenylene sulfide (PPS). Good results were also obtained with nylon and polycarbonate, while tests with PEEK were totally unsuccessful. However, the lack of success with PEEK was, apparently, the result of test rig hardware limitations and not a fundamental conceptual limitation of the process. In general, it is felt that the process is applicable to any thermoplastic material which has a sufficient processing temperature window to be processed by commercial extrusion or injection molding equipment.

The following paragraphs describe the results obtained and what was learned that can be useful for further development.

1. <u>Wet-out varied significantly from material to material.</u> The most noticeable performance variation from material to material was in the area of wet-out. PPS showed the most aggressive wet-out characteristics while PE showed the least tendency to wet-out the entire tow. This translated to a higher degree of resin penetration with PPS than with PE. On samples which were knife cut in the unmounted state, individual fibers or groups of fibers typically separated from the resin in the PE samples, indicating a lack of adhesion. On the other hand, the PPS impregnated samples typically fractured through the resin with individual fibers retaining some resin coating when knife cut in a similar fashion.

2. <u>Temperatures, rather than pressures, appear to be the primary process variables controlling the quality of impregnation.</u> - During experimental runs, wide swings in both coating temperatures and pressures were intentionally made. While there appeared to be an optimal temperature point for maximizing impregnation quality, no optimal pressure level was found. Furthermore, it was not possible to compensate for high resin viscosity caused by low temperature by raising the pressure. The wet-out characteristics appear to depend on capillary action between the molten resin and the surrounding fibers, much like a metallic solder joint. This capillary effect explains why increased pressure cannot compensate for low temperature.

   If the coating block temperature was too low, the block would not fill with resin and the center of the tow would be starved. The indicator of a sufficiently high block temperature was the weeping of molten resin from the vent hole located upstream of the tow inlet port.

If the coating block temperature was too high, the viscosity of the resin would drop to a point where regions of pure resin would coextrude along side the fiber bundle. This coextrusion would occur whether or not the tow was moving.

Under optimum temperature conditions, no resin would exit from the outlet port unless pulled by the tow and the bulk of the resin would be distributed throughout the tow. Optimum temperatures appeared to be similar to recommended extrusion temperatures and somewhat lower than supplier recommended injection molding temperatures.

3. <u>The required coating block pressures appear to be about 1000 psi.</u> - While no means was available for directly measuring the internal pressures in the coating block, the pressure on the injection press air cylinder was measured and controlled. Typically, the press air temperatures ranged from 30 to 40 psi. With a 50:1 pressure multiplier, this would equate to 1500 to 2000 psi on the resin at the top of the molding press, assuming a frictionless system. It seems likely that 30 to 40 percent of the pressure would be lost to friction, resulting in a pressure between 900 and 1400 psi in the coating block.

   In a more sophisticated system, it would be advisable to measure both the cavity temperature and pressure directly.

4. <u>Tow preheat is an important factor in the overall system design.</u> - In many ways, the melt impregnation approach is analogous to "tinning" of stranded electrical wire with solder. The quality of the impregnation is dependent on the temperature of the strands being impregnated. Unfortunately, glass fibers are poor conductors of heat and a significant amount of thermal energy must be put into the fibers before or during the impregnation process.

   A heat balance calculation for a PPS impregnated tow with a 62 percent volume fill of glass fibers shows that almost two thirds of the heat addition required is to the fibers rather than to the resin. Since the impregnation processing must take place near the melt temperature of the resin, it is impossible to add sufficient energy through the resin to bring the tow up to temperature without degrading or destroying the resin. This problem is especially severe with the high performance resins like PEEK where there is little difference between the melt temperature and the degradation temperature. For materials like polyethylene, the problem is much less severe.

On the benchtop system, a hot air tow preheater was used which consisted of a four foot length of tubing with the tow being passed through counterflowing hot air with an inlet temperature in the 700°F range. The temperature achieved by the individual strands at the center of the tow was, of course, variable, depending on the tow feed rate and any starts/stops which might occur. Based on qualitative observations, it appeared that a much longer preheat chamber with a lower air inlet temperature would be very beneficial.

With the existing test rig, there was an obvious difference between the resin penetration on a tow which had been stopped in the preheat tunnel and material which had passed through at high speed. Material which had been stopped in the tunnel would show discoloration due to degradation of the sizing on the glass and would also show a greater affinity for wetting out with resin. Material which had been pulled through rapidly would likely show a resin starved center core, even though the coating block remained full of resin.

As would be expected, the importance of tow preheat with polyethylene resin was much less than with the engineering resins like PPS and nylon. Since polyethylene can be heated well above its melting point without degradation, it is possible to use less tow preheat and still have sufficient heat available to get resin flow during the impregnation process.

5. <u>Processing of PEEK was not possible on the existing test rig but might be possible on a system with better control</u> - When attempting to run PEEK through the system, material degradation and blockage in the injection press ram area occurred. This blockage occurred upstream of the coating block and, apparently, would have occurred even if the block had been removed.

   Since PEEK is commercially processed by both injection molding and extrusion without exotic equipment, it seems that the problem was related to the specifics of the molding press, rather than inherent limitations of the material. However, it is also clear that an extremely small temperature window exists between the point where PEEK will flow and the point at which it degrades. It is also obvious that it remains a very viscous material when it does melt. These factors may make it very difficult to use for melt impregnation, even with a more sophisticated system.

The primary set of test runs were first discussed in Progress Report NCI 93-02. The key observations reported at that time are repeated below.

Tests were conducted with PPS resins in two flow grades supplied by Hoechst Celanese under their designations Fortron 0203B6 and 0205B4. Fibers tested included Owens-Corning S Glass with an epoxy sizing and PPG Industries' E glass which was unsized. Both were nominal 250 yard/pound materials. Under the microscope, the PPG Industries' material appears to have a larger individual fiber diameter and a lower total fiber count. A variety of samples were mounted in epoxy resin and polished for examination. The samples examined showed that there were varying degrees of nonuniformities depending on the materials and the processing conditions.

Mechanical handling problems were encountered with the unsized E glass when the tow was threaded through the tension pins, which had been added to the resin cavity to promote fiber spreading. While the E glass sample from PPG Industries had a coating on it to aid in mechanical handling, it was found that it had a much greater tendency to shred and jam when fed through the tensioning pin system than did the Owens Corning S glass supplied by MTL. The E glass was eventually abandoned and efforts were concentrated on the S glass and the fibers were fed over the top of all three of the tensioning pins, rather than in an over/under/over pattern to reduce jamming.

Exhibits VI and VII, on the following page, are typical of the 100X photographs which were obtained with PPS resin and the S glass. Thirty percent fiber volume fraction is estimated, or about half of what would be needed for maximum performance material. This resin rich consistency is largely a result of the breadboard resin feed system (based on a laboratory injection molding press) which controls pressure rather than resin flow. Several points are worth noting from the exhibits:

- First, there is a noticeable "grain" of high fiber density material in each of the samples. This grain geometry equates to the strand size in the unimpregnated tow. In the majority of the samples, the strands maintained some integrity, even though resin penetration completely through the strand is occurring.

- There are some areas of uncoated fibers, indicated by the dark areas on the photographs.

- In general, the fiber strands nearest to the outside edges tended to break up into individual fibers and the graininess disappears. This indicates that a thin ribbon-like geometry, where a much greater percentage of the fibers are at, or near, the surface, may dramatically reduce the grain patterns.

- The dimensions of the resin rich areas are similar to the strand diameters, indicating that reduced strand diameter might reduce resin rich areas.

100X

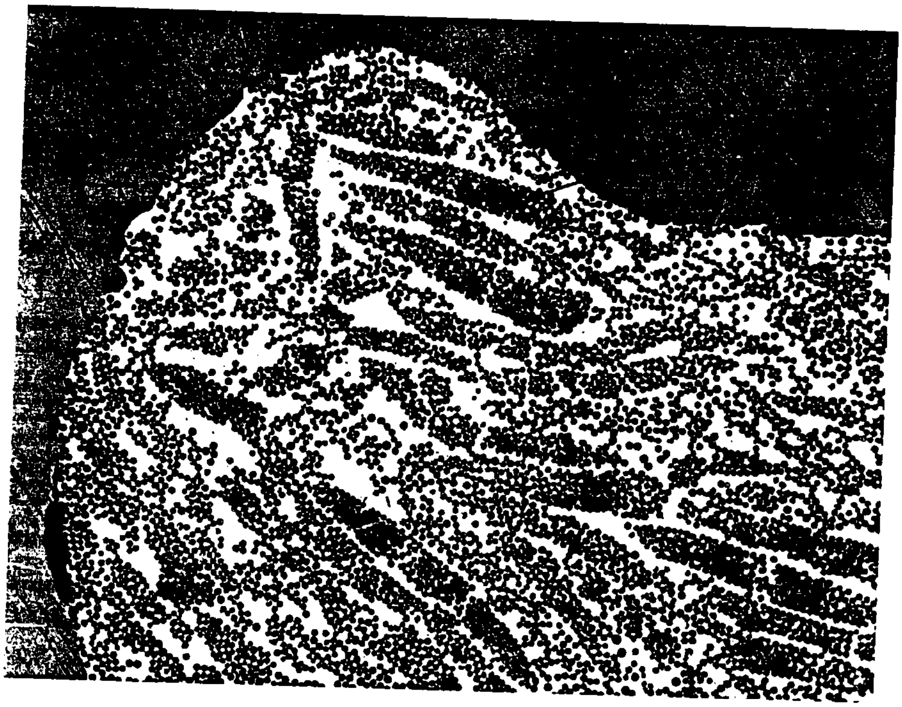


EXHIBIT VI - PPS AND S GLASS - PHOTO 1 AT 100X

100X

EXHIBIT VII - PPS AND S GLASS - PHOTO 2 AT 100X

Exhibits VIII and IX show two examples of material without graininess. Exhibit VIII is E glass with PPS resin and Exhibit IX is S glass with PE resin. Both are at 100X magnification. Several points can also be noted from these samples.

From the E glass/PPS sample (Exhibit VIII):

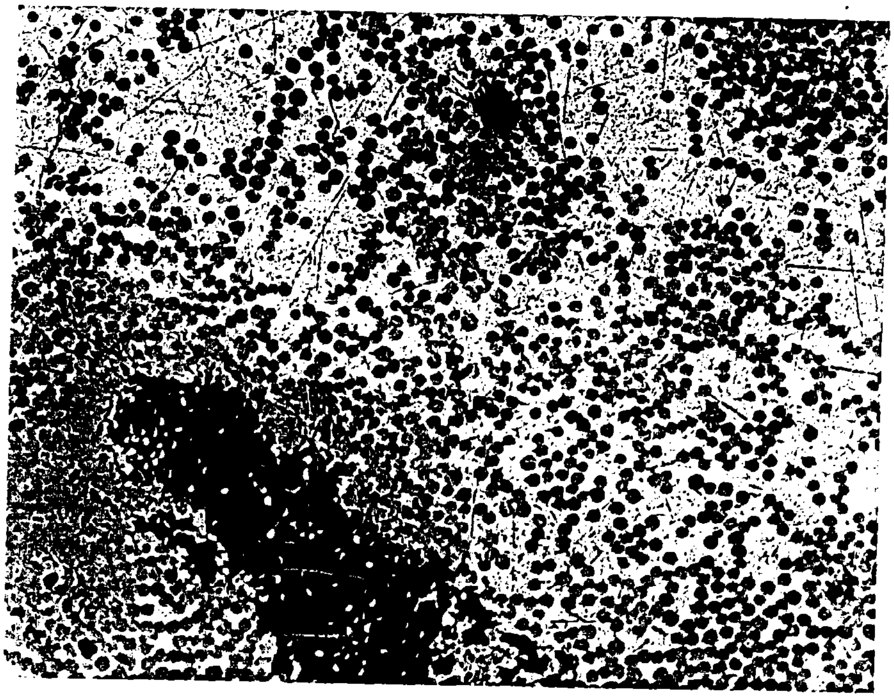
- o The lack of graininess in the E glass sample can be attributed to the fact that the E glass samples were a single unstranded tow in which all fibers were held together into a single ribbon.
- o The E glass sample also had major nonuniformities in the resin distribution with large areas of uncoated fibers and also large resin rich areas.

From the S glass/polyethylene sample (Exhibit IX):

- o This sample shows a lack of graininess even though it was run with the S glass tow which produced graininess with PPS. With the PE, it was possible to thread the tensioning pins in an over-under-over fashion, flexing the tow to disassemble the strands and allow the resin to flow into the interfiber spaces.
- o There is a relatively high percentage of voids because PE does not wet out as well as PPS,
- o In the impregnated regions, the fiber/resin ratio appears to be quite high. However, with polyethylene, the system produced coextruded streams of pure resin in which an estimated 30 percent of the total cross section would contain no fibers at all.

In addition to the melt impregnation tests described above, limited tests were conducted with a roller squeegee post-melt processing system. The idea was to squeeze and reflow a semi-impregnated tow in a post impregnation operation. Conceptually, such a system could remove the grain structure discussed above. It was also expected that it would be possible to process the uncoated tow through the rolls, forming a sandwich with thin resin tapes as an alternative to the melt impregnation approach.

Unfortunately, the results were not at all encouraging. It was found that the tow, whether precoated or uncoated, tended to mechanically jam in the roller gap periodically as twisted strands fed through the gap. Conceptually at least, this problem could be compensated for by spring loading one of the rollers. A second problem encountered was that the tow does not want to track straight through the rollers and tends to wander from side to side. This problem might be fixed by using a grooved roller, but a groove might aggravate the jamming problem. In summary, it is felt that post processing by rollers is, not a viable idea for "simple in-line" systems.



100X

EXHIBIT VIII - E GLASS WITH PPS RESIN - PHOTO 3 AT 100X

100X

EXHIBIT IX - S GLASS WITH POLYETHYLENE - PHOTO 4 AT 100X

V. CONCLUSIONS

The relatively simple qualitative tests conducted during Phase I have demonstrated that the melt impregnation approach for producing thermoplastic tow on-line is technically feasible and should be pursued during a Phase II activity. At the same time, the early testing has demonstrated that the process does have some critical development needs and may have some fundamental limitations.

The following are the four primary conclusions which have been drawn during this Phase I study.

1. <u>Coating speeds for a production scale in-line melt impregnation system are likely to be significantly below thermoset coating speeds.</u> - While coating speeds during the Phase I benchtop tests were typically in the range 1 to 5 feet per minute, it is believed that speed was limited more by preheat tunnel length than by any other factor. With an adequate tow preheat tunnel length, it should be possible to substantially increase this speed, but it is unlikely that it will be possible to reach speeds common to thermoset filament winding.

2. <u>Much of the required melt processing technology can be adapted from commercial extrusion equipment.</u> - While inline melt impregnation represents a "new to the world" application, the technologies of preparing, controlling and metering the melt are similar to approaches used for commercial extrusions equipment. Specific pieces of applicable technology include:

   - Multizone temperature control

   - Extruder screw and drive design

   - Pellet/powder feed systems, and dryers.

   For a prototype in-line impregnation, a commercial extruder is a logical starting point.

3. <u>The in-line impregnation system could be carriage mounted on a filament winder, but it is likely to be a sizeable device.</u> - The overall size of a melt impregnation module may be estimated at approximately 60 inches high by 18 inches deep by 12 inches wide. The weight of the unit would likely be between 100 and 200 pounds. While a device of this size could be mounted on a traversing slide system, it is likely to be somewhat oversize to mount on an existing filament winder guide system and may require a separate set of support/guide rails.

4. <u>A minimum practical size for an in-line impregnation module is a system which combines about six tows into a single tape or strand.</u> - For all practical purposes, it should be as easy to impregnate multiple tows as it is to do a single tow. The melt rate required to impregnate a single tow is too low to fully utilize the output of even the smallest commercial extruder. A 1/2 inch diameter screw is about the smallest size widely available. Such a screw can supply sufficient plastic to impregnate several tows at normal speed. If the screw speed is reduced, the residence time of the plastic in the extruder increases and the material tends to degrade. Going to a smaller diameter screw in the extruder greatly reduces the torque capacity of the device. Thus, the melt section of a six tow system can be built more easily than the melt section for a single tow system. Of course, a multi-tow preheat system must be developed.

The most workable output format for most applications would be a flat tape. Based on the tow geometry used in Phase I, a tape width of about 0.25 inch and a tape thickness of about 0.040 inch would be produced.

If preferred for specific applications, the output format could also be a round rod. This geometry would hold heat longer and might, therefore, be easier to filament wind and reconsolidate after winding.

VI. RECOMMENDATIONS

It is recommended that the development of an in-line impregnation module for thermoplastic composites be continued with the design and construction of a prototype system capable of impregnating multiple tows with polyphenylene sulfide (PPS) or other engineering resins. The system should be designed for use with an existing commercial (thermoset) filament winder for cost reasons.

The prototype system should include the following subsystems:

- Tow supply subsystem
- Tow preheat subsystem
- Melt supply subsystem
- Impregnation module
- Carriage subsystem and winder interface

Of these subsystems, the tow supply and the melt supply subsystems can be based on semi-standard commercial equipment or components while the remaining subsystems must be designed and built from the component level.

Once the prototype has been designed and built, it should be tested with a range of resins which may be of interest to users of filament wound structures within the Army. These applications may include a range of materials from recycled commodity thermoplastics to engineering resins such as PPS.

North Coast Innovation is currently in the process of identifying specific end uses. Three potential Army applications already identified include the following:

- Damage tolerant (impact resistant) rocket motor housings which take advantage of advanced thermoplastics (MICOM).
- Self protecting munitions housings/containers which will melt in case of accidental fire to limit pressure buildup (MICOM).
- Corrosion resistant construction forms made from filament wound tubes, possibly made with recycled thermoplastic resins (CERL, Army Corp of Engineers).

North Coast Innovation will be submitting a proposal, under separate cover, for the development of the prototype system as a Phase II SBIR effort.

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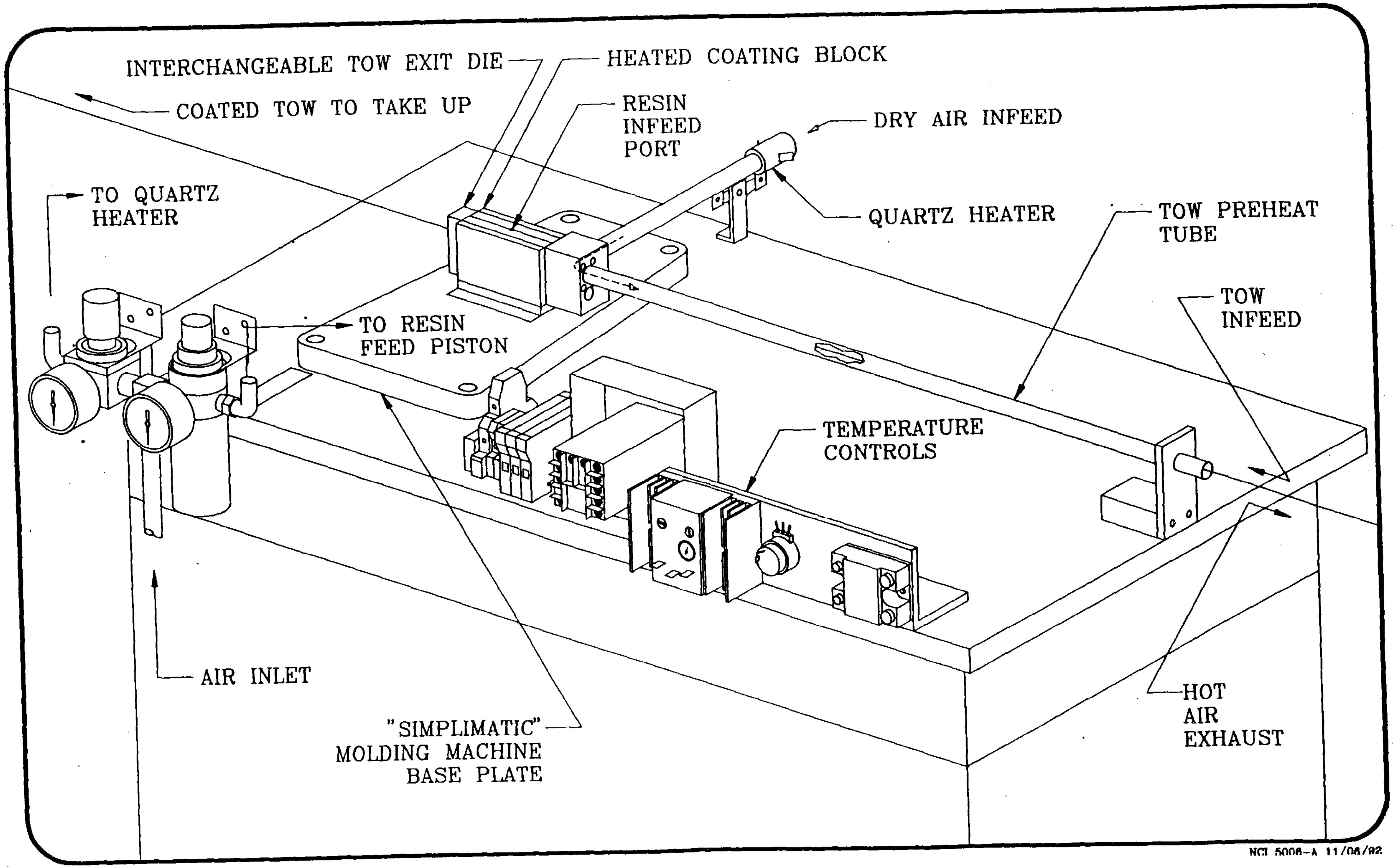
APPENDIX A

DRAWINGS/SKETCHES OF KEY COMPONENTS



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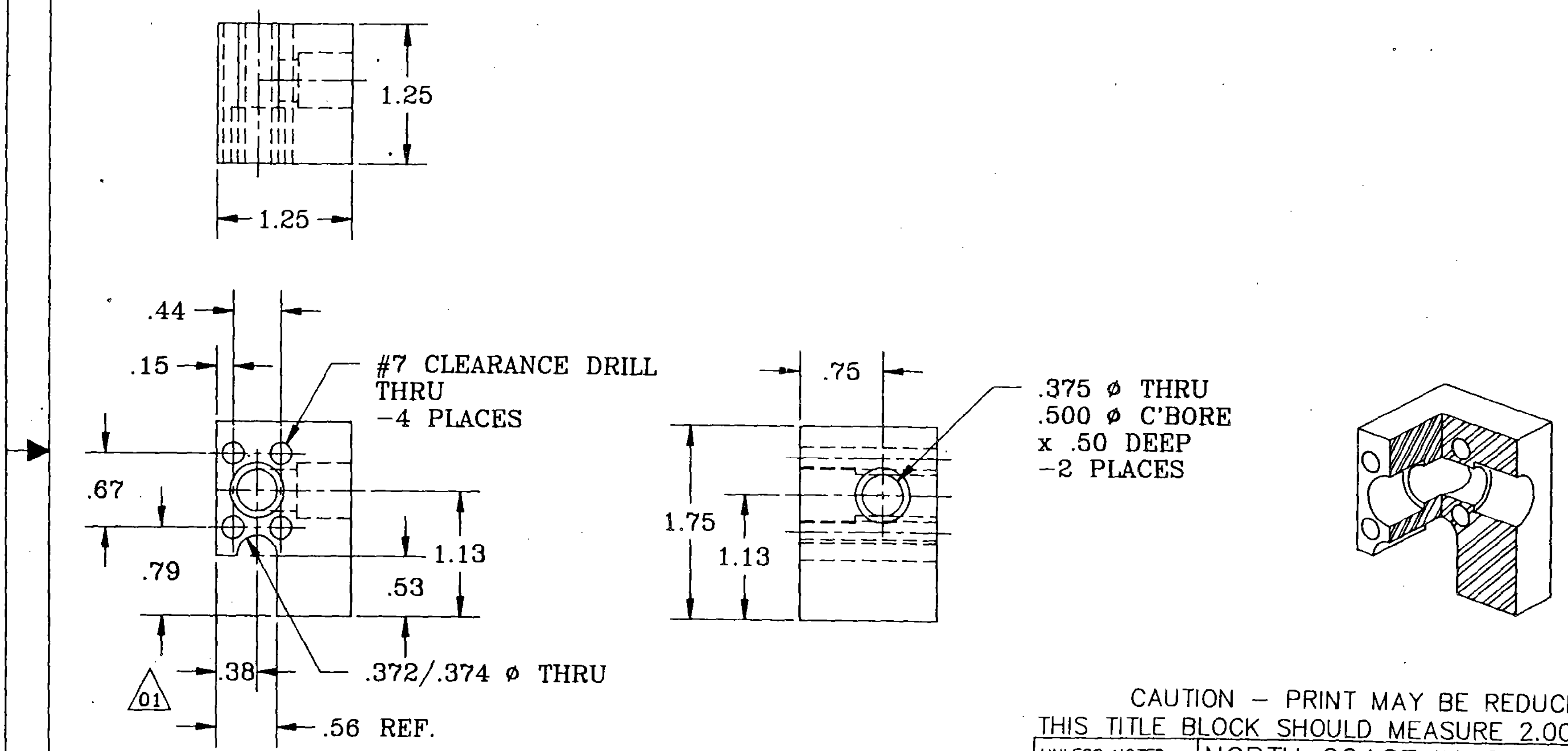

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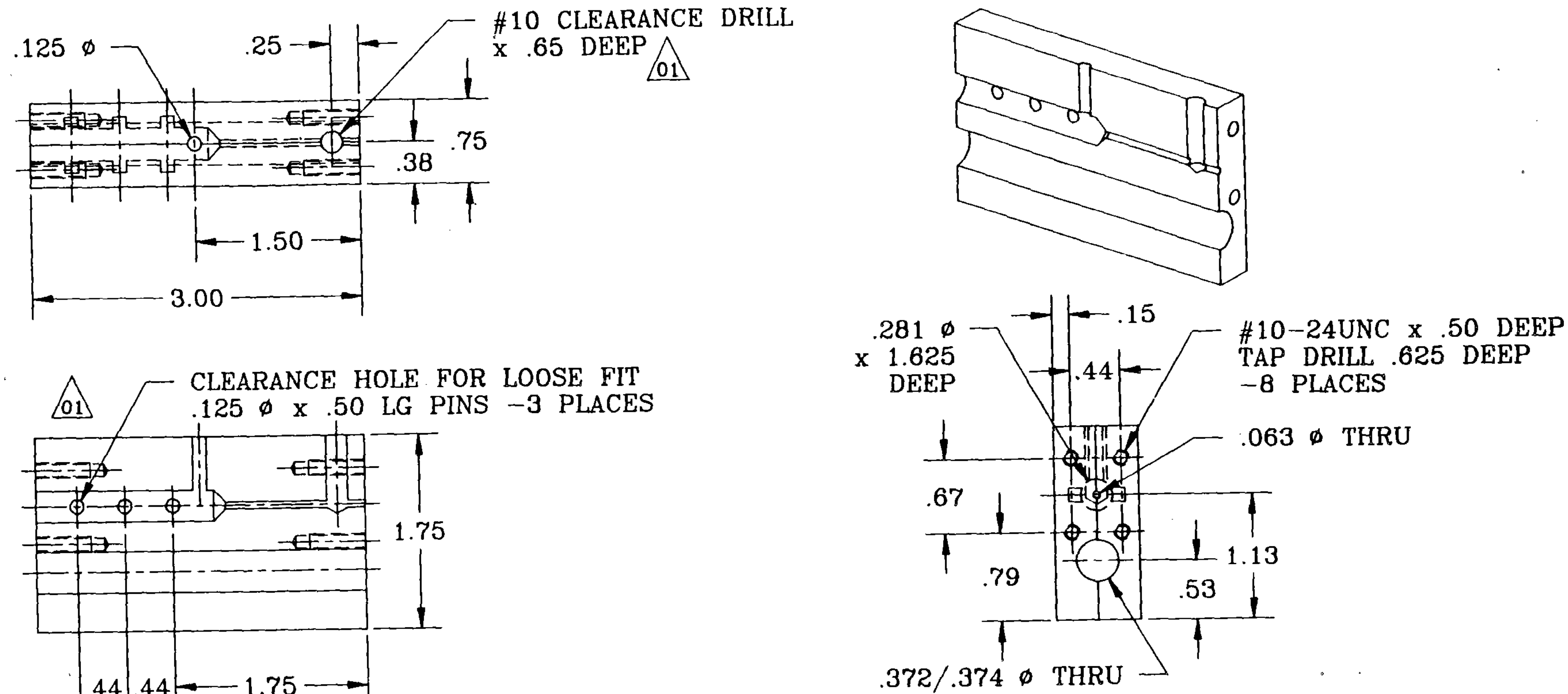

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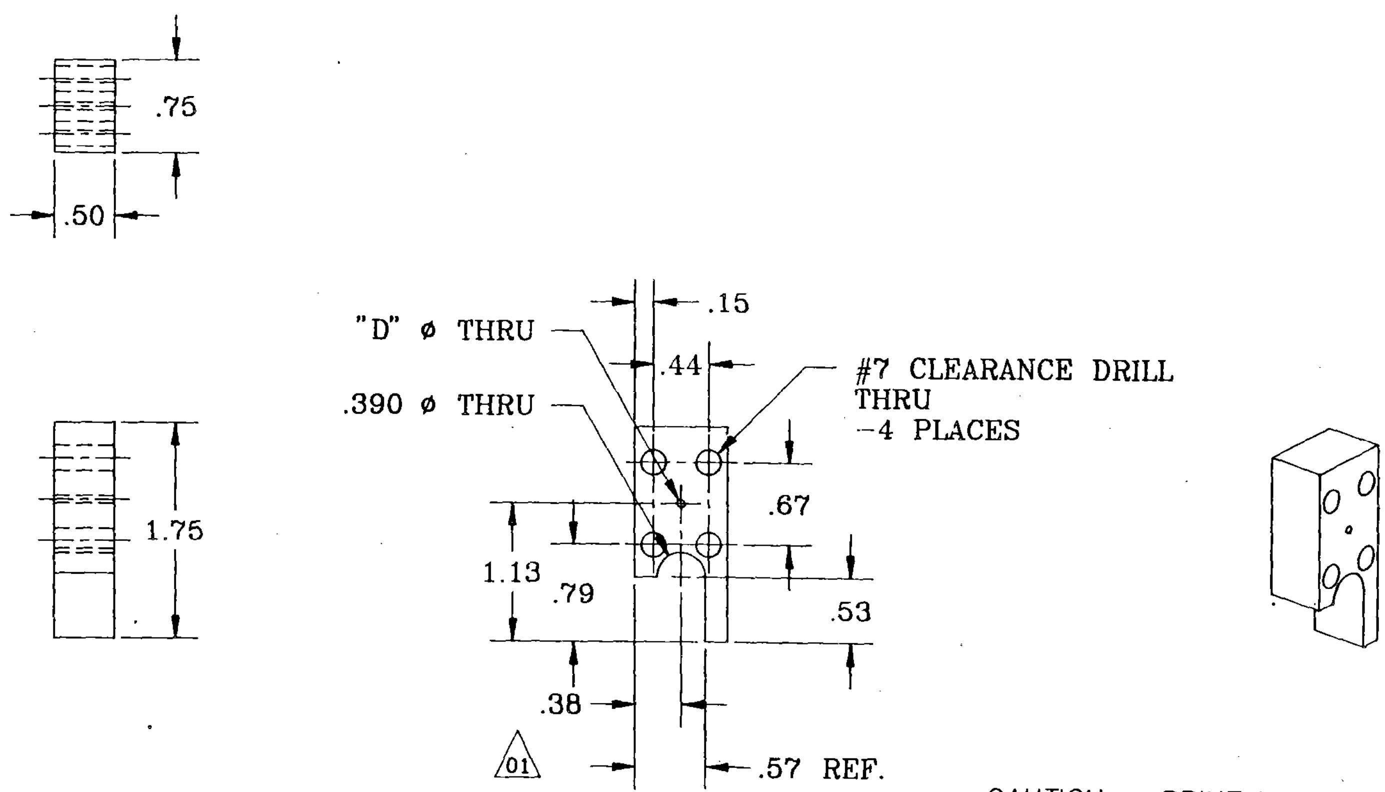

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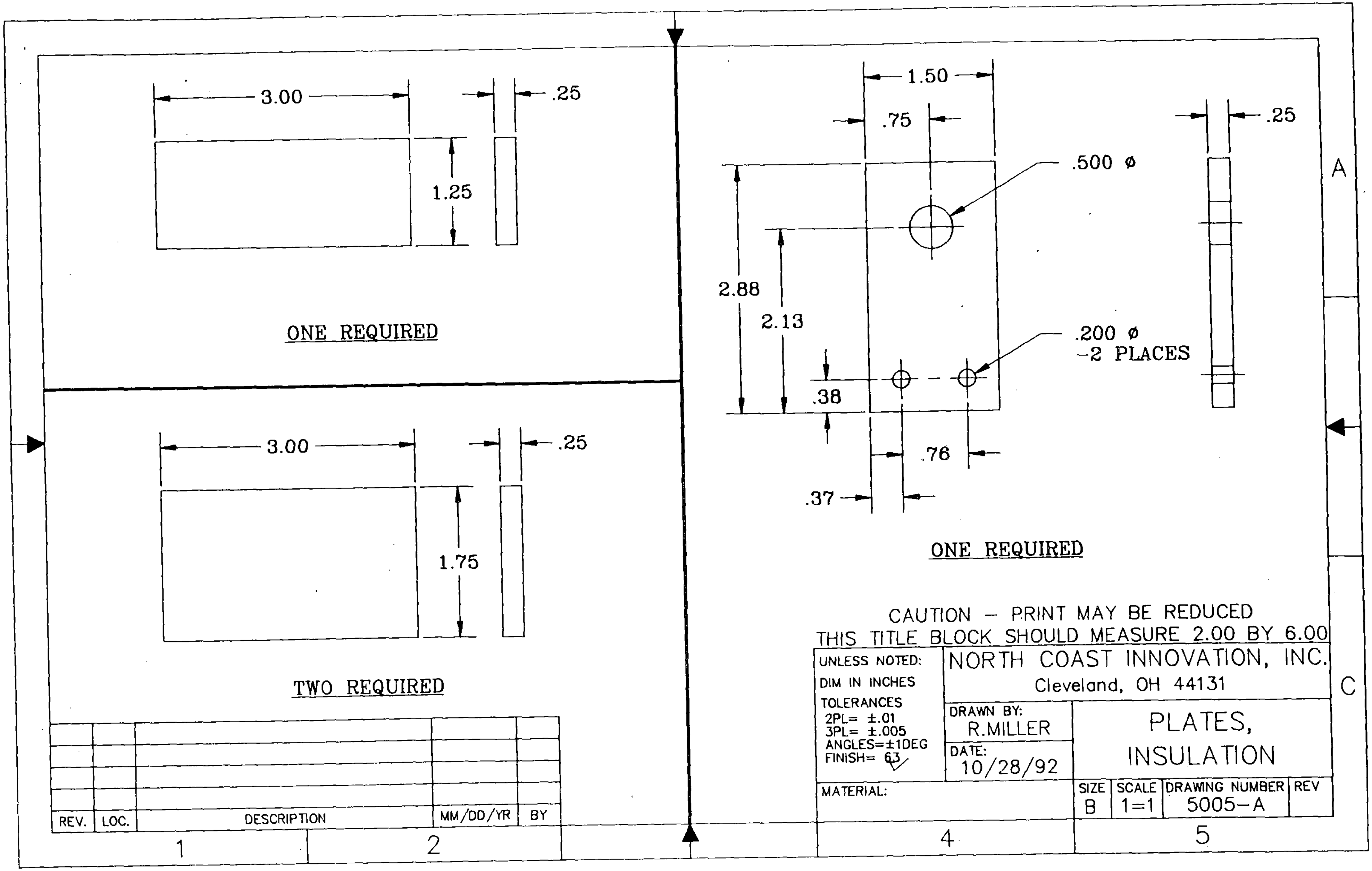
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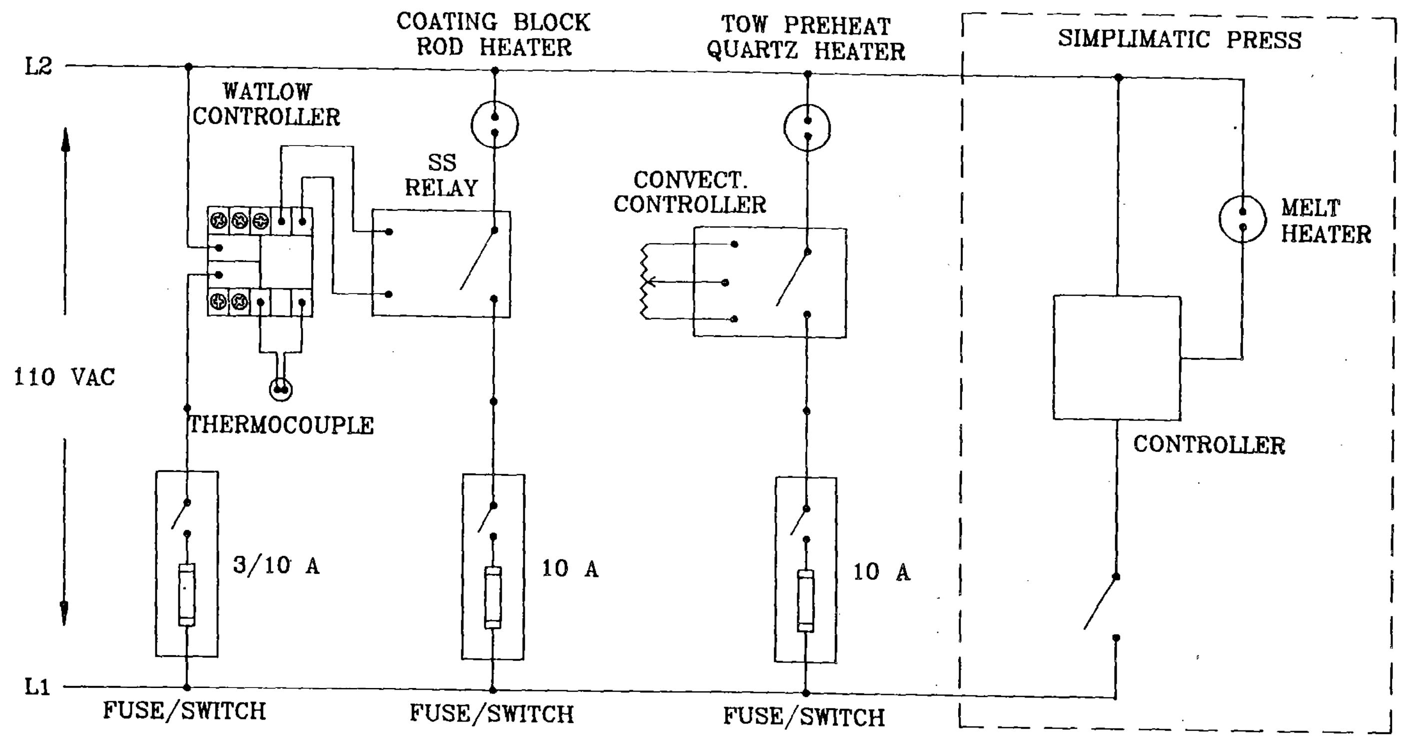

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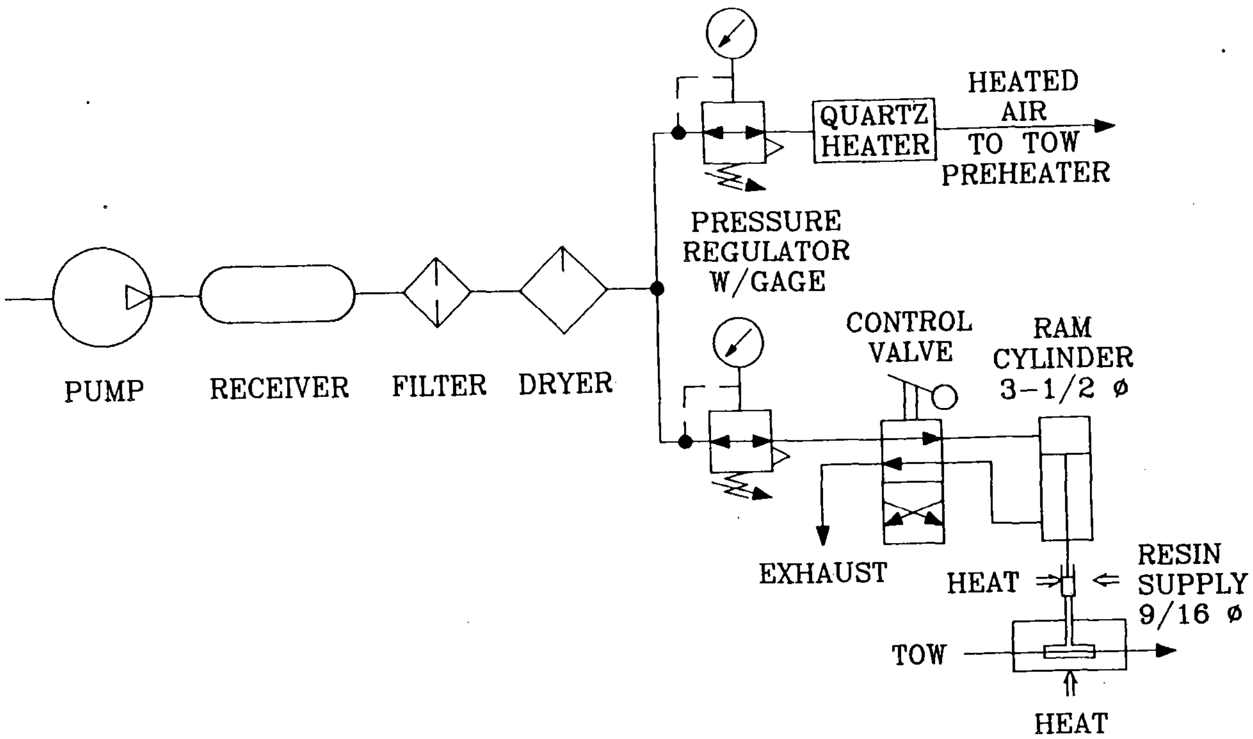

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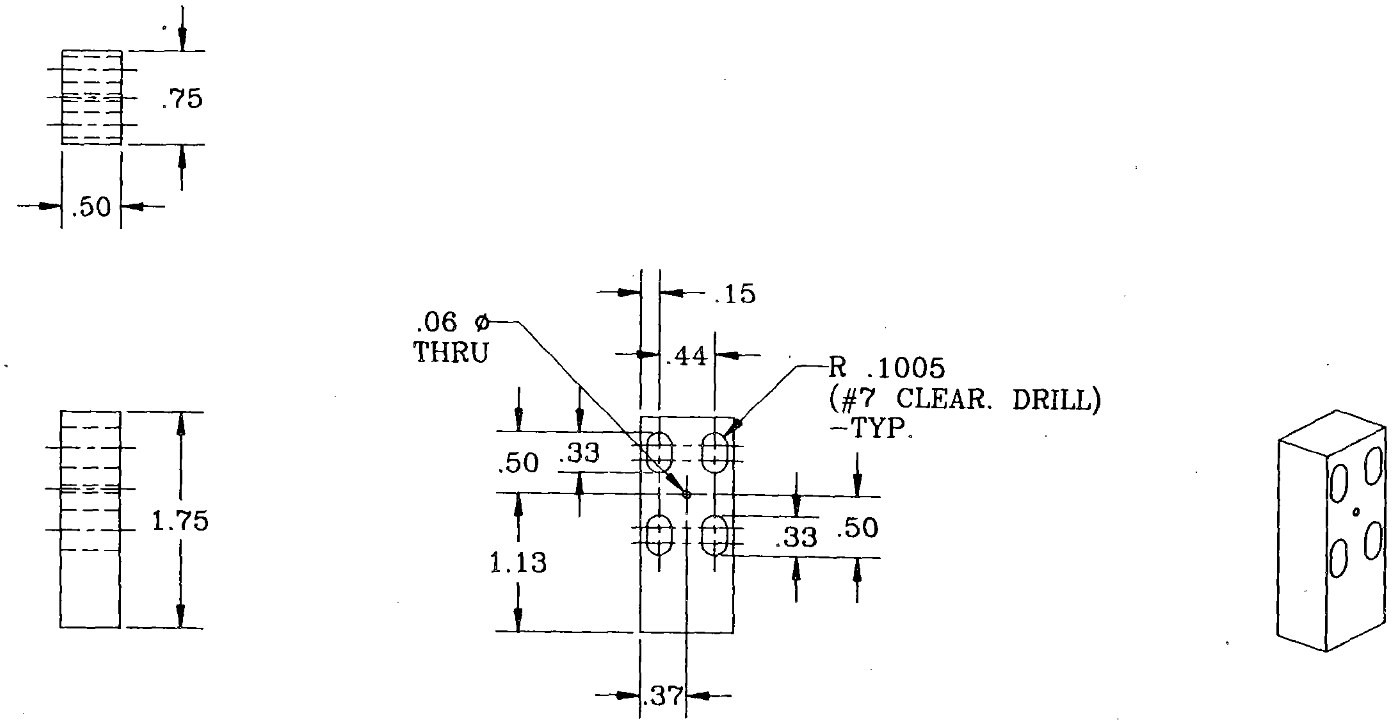

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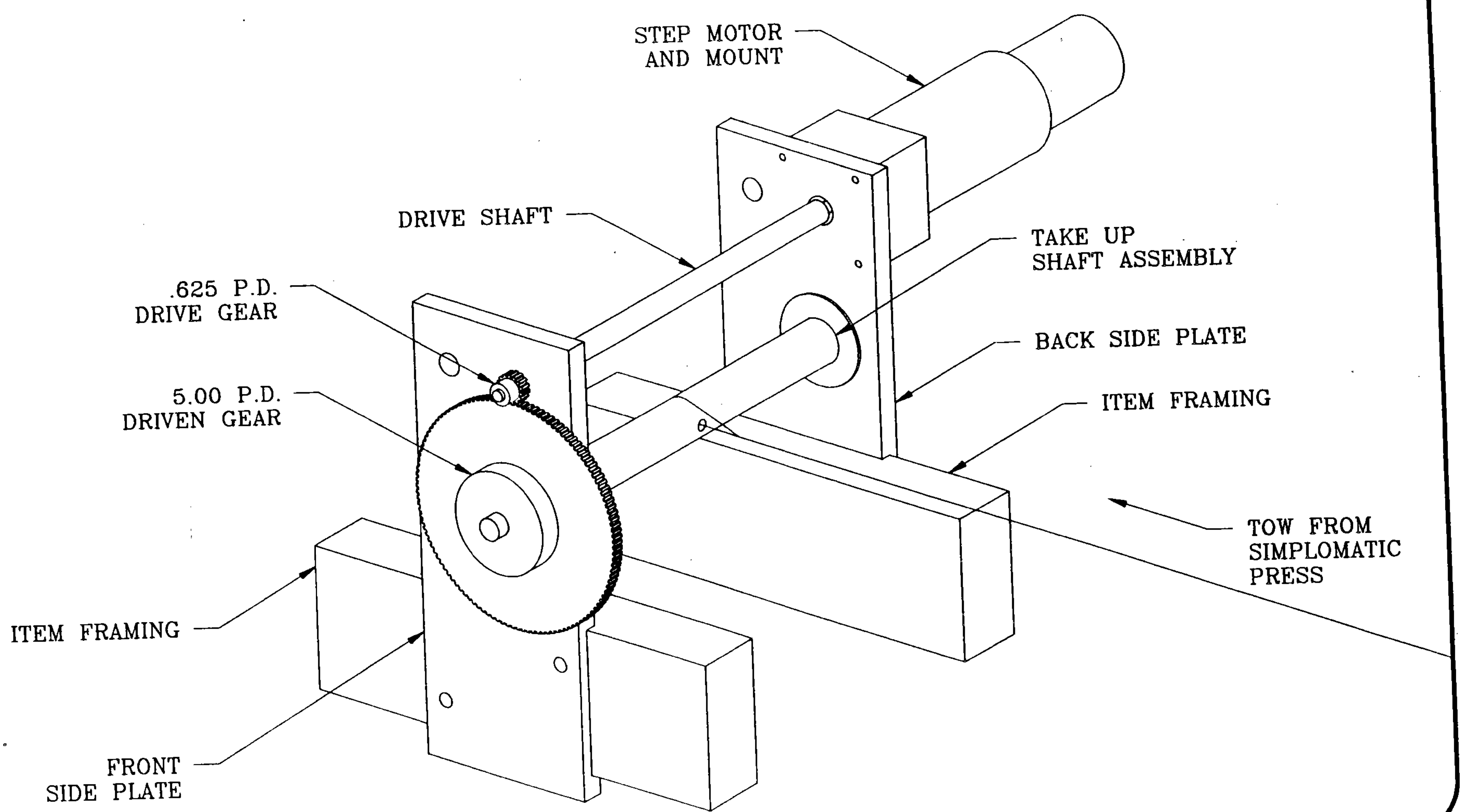

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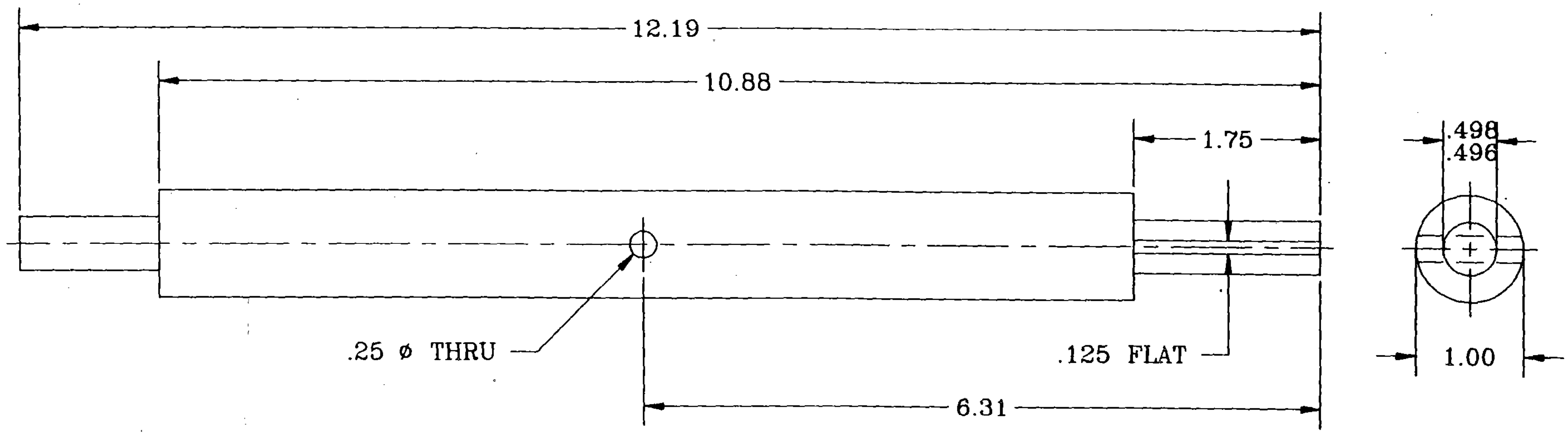

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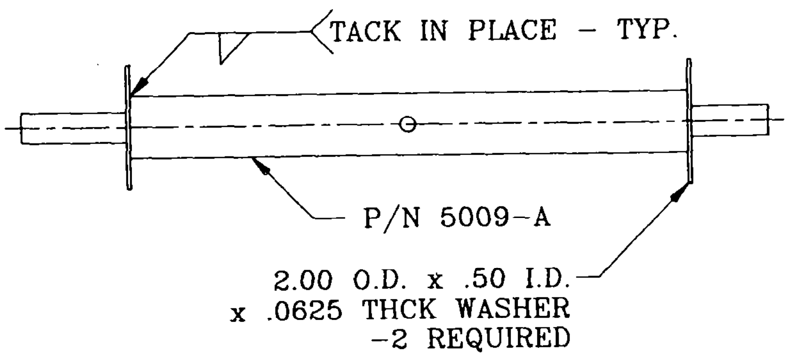

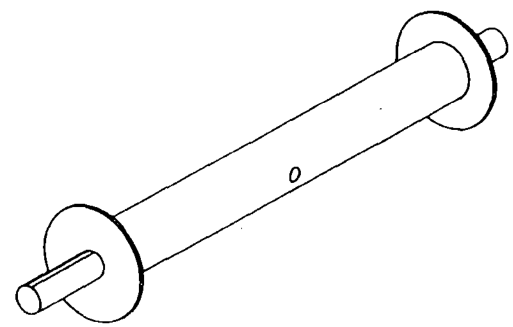

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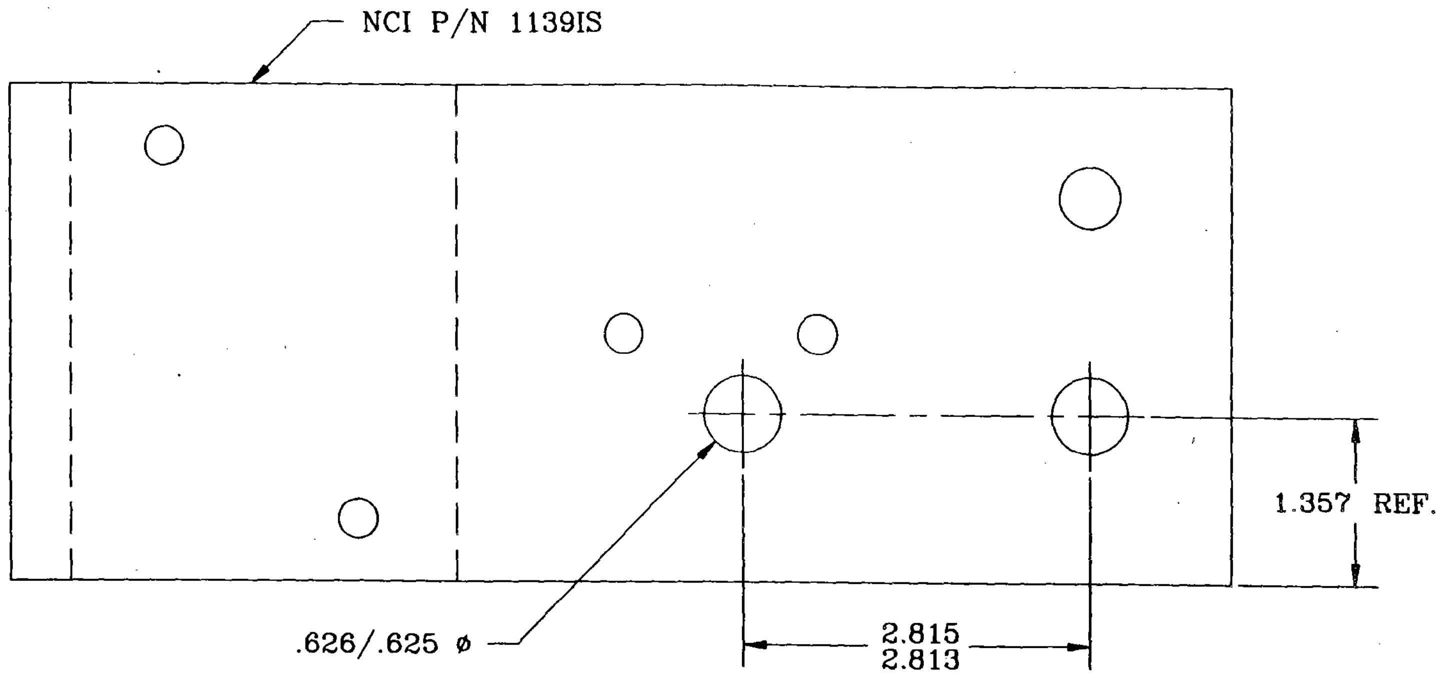

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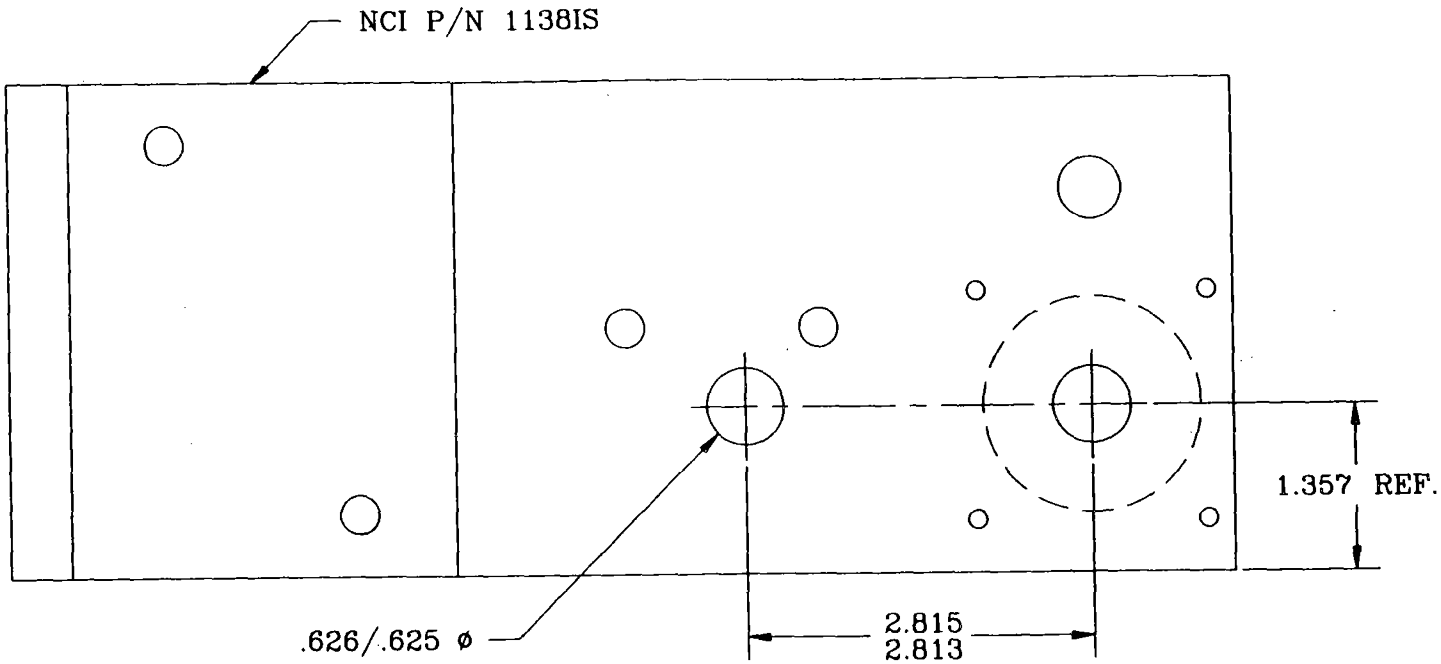

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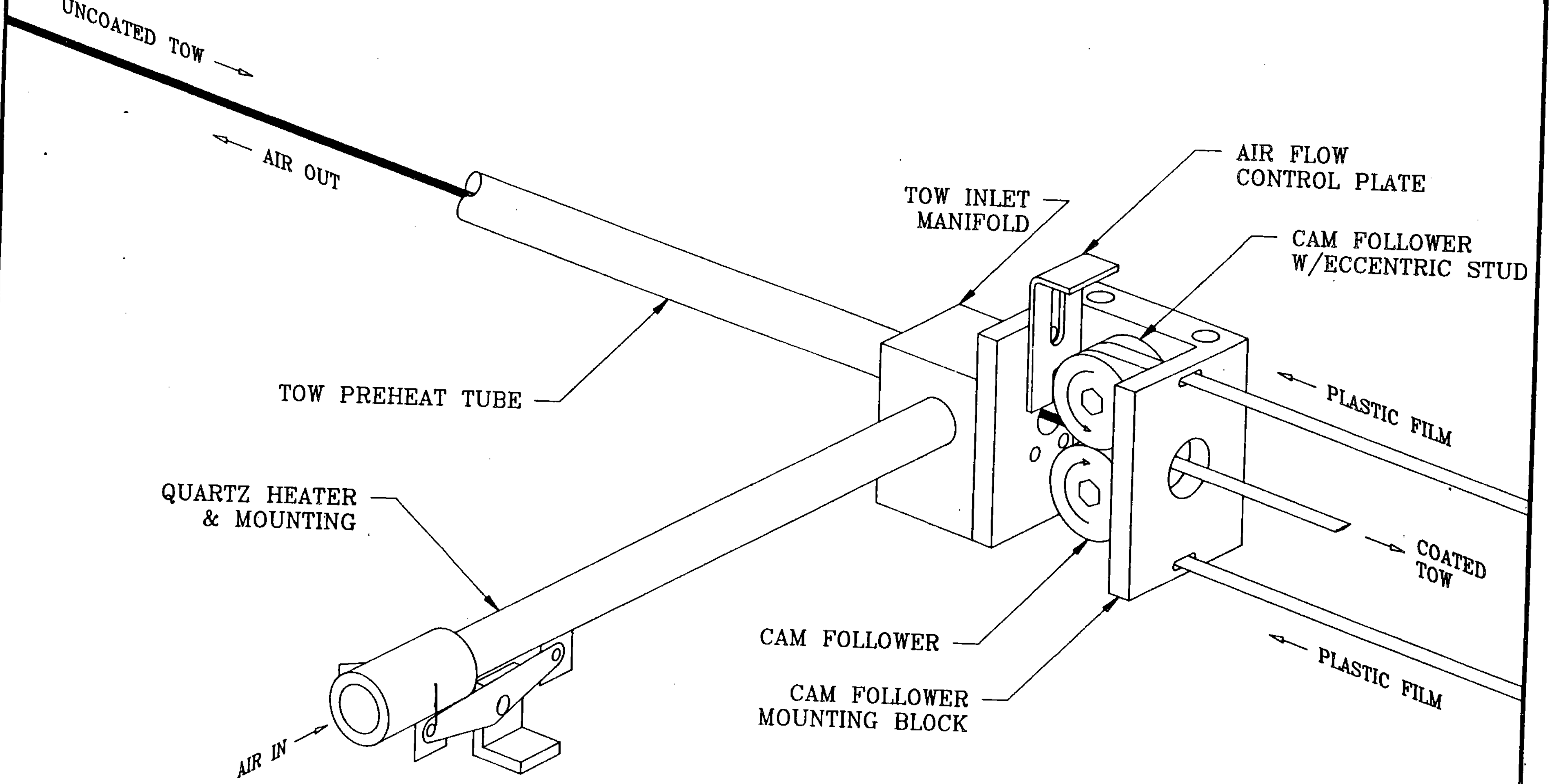

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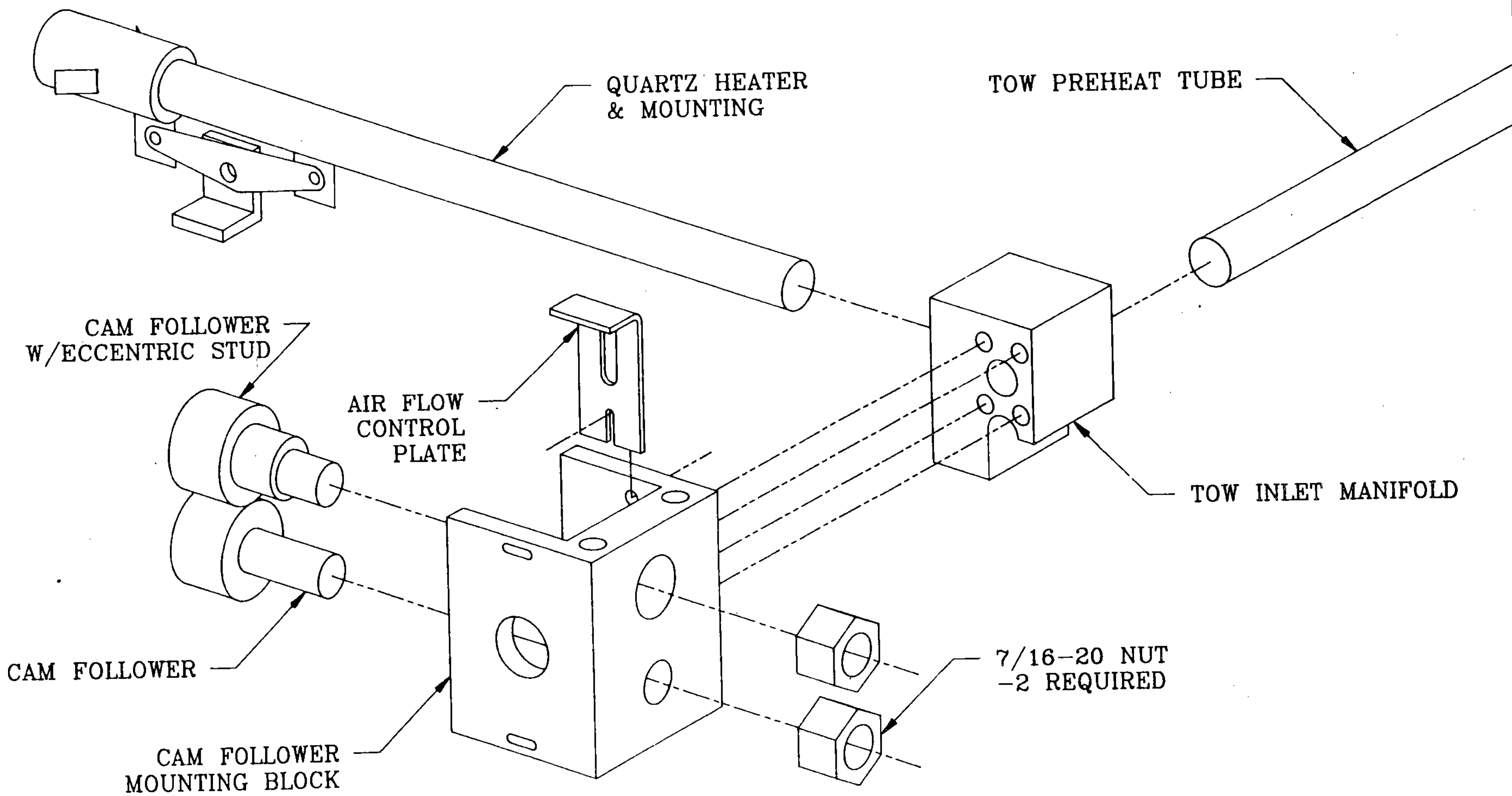

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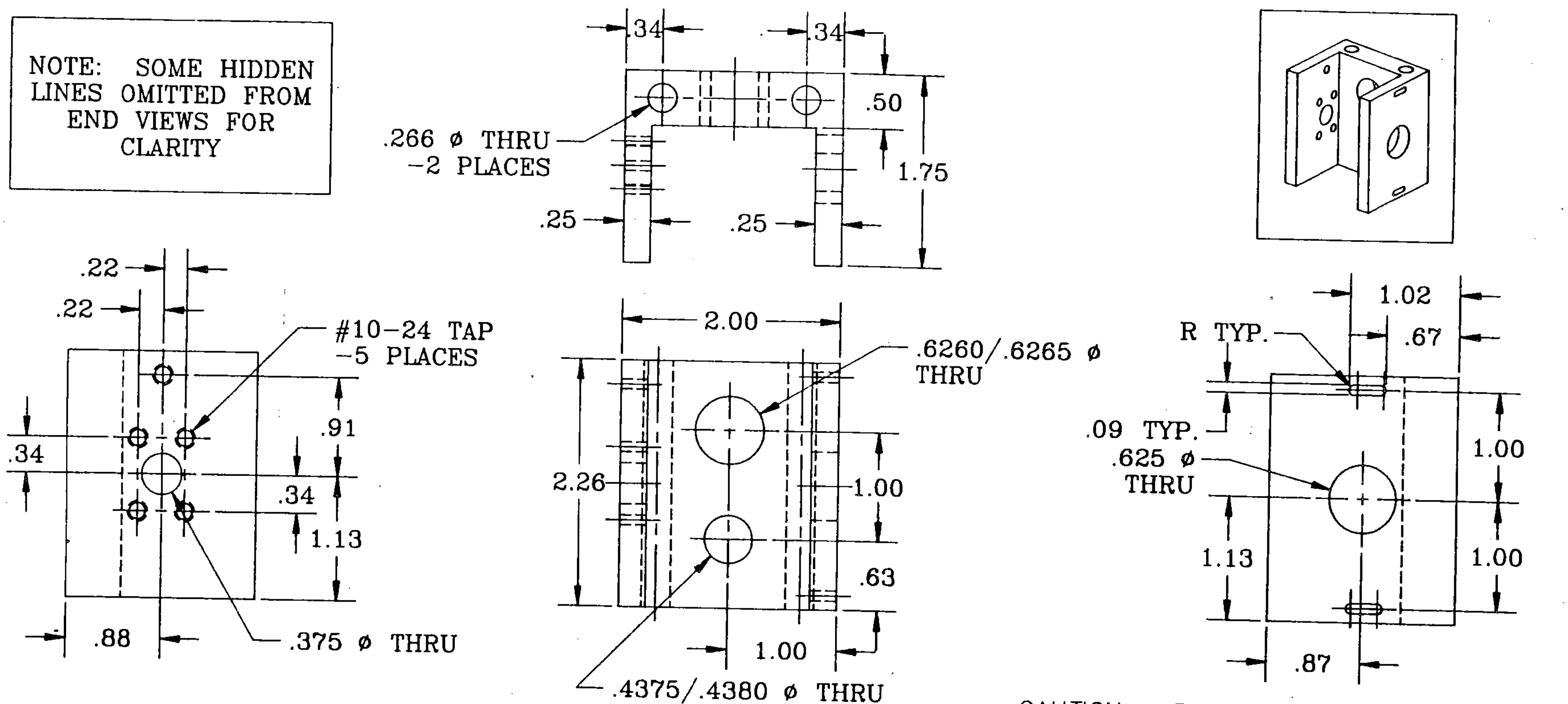

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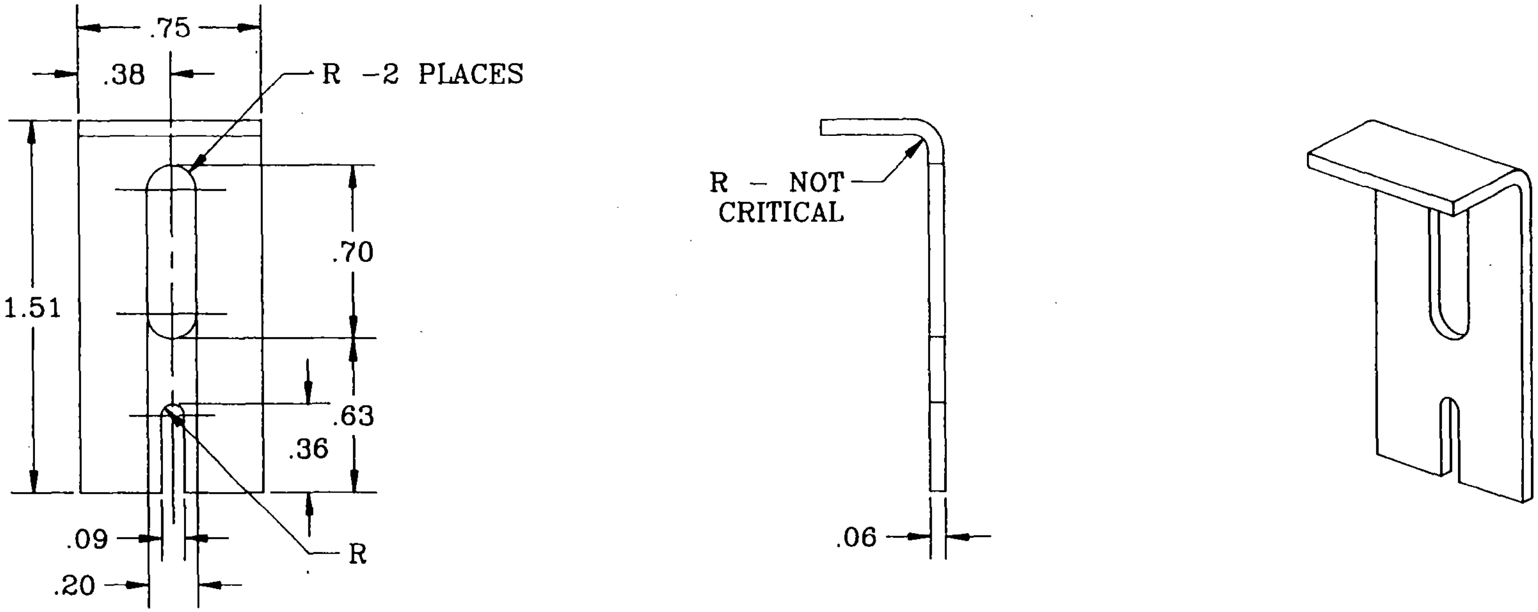

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1	NASA - Lewis Research Center, 2100 Brookpark Road, Cleveland, OH 44135-3191
	Director, Defense Intelligence Agency, Washington, DC 20340-6053
1	ATTN: ODT-5A (Mr. Frank Jaeger)
	Director, U.S. Army Research Laboratory, Watertown, MA 02172-0001
2	ATTN: AMSRL-OP-CI-D, Technical Library
1	AMSRL-OP-CI-D, Visual Information Center
1	AMSRL-OP-PR-WT
15	AMSRL-MA-PA, Seth Ghiorse, COR